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16. Abstract

The effect of moisture on the fatigue behavior of several multidirectional graphite/epoxy composite laminates has been investigated. In compression-compression fatigue (R = 10) studies performed on $[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$, T300/934 laminates, the fatigue life, N_f, at a given stress amplitude, and the apparent fatigue limit, at 5×10^6 cycles, are shown to be significantly reduced by the presence of from 1.4 to 2% moisture. The form of the moisture distribution in the specimen (gradient and flat profile) was considered to establish the influence of accelerated moisture conditioning on fatigue behavior. For the gradient specimens having an average moisture content of 1.4%, N_f was reduced by a factor of 8 at all stress levels investigated. Corresponding reduction in N_f for the flat moisture profile specimens at the same average moisture content was comparatively smaller, being about a factor of 5 from the value in dry specimens. X-ray radiographic analysis of damage accumulation in compression-compression fatigue revealed interlaminar cracking to be the dominant mode of failure responsible for the observed enhanced cyclic degradation of moisture-conditioned specimens. This finding is corroborated by the observed systematic reduction in interlaminar shear strength as a function of moisture content, which, in turn, increased the propensity for delamination under cyclic compressive loads. Residual strength measurements on cycled specimens indicate significant strength reductions at long lives, particularly in moisture-conditioned specimens.

Tension-compression fatigue studies conducted on a $[0/\pm30]_{3s}$ AS/3501 laminate over a range of negative stress ratios indicated that a strong moisture-induced degradation in fatigue properties can occur even in the presence of moderate compressive components (20% of the total stress range). The observed reductions in N_f in this case were found to be related to the early initiation of fatigue damage in moisture-containing specimens. In tension-tension fatigue of the T300/934, $[0/\pm45/90]_{2s}$ laminate, moisture was found to enhance degradation only slightly. Here, it appears that the influence of moisture is to intensify the susceptibility of this laminate to edge delamination.

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OF GRAPHITE/EPOXY COMPOSITE LAMINATES

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ABSTRACT

The effect of moisture on the fatigue behavior of several multidirectional graphite/epoxy composite laminates has been investigated. In compression-compression fatigue (R = 10) studies performed on $[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$, T300/934 laminates, the fatigue life, N_f, at a given stress amplitude, and the apparent fatigue limit, at 5×10^6 cycles, are shown to be significantly reduced by the presence of from 1.4 to 2% moisture. The form of the moisture distribution in the specimen (gradient and flat profile) was considered to establish the influence of accelerated moisture conditioning on fatigue behavior. For the gradient specimens having an average moisture content of 1.4%, N_f was reduced by a factor of 8 at all stress levels investigated. Corresponding

reduction in N_f for the flat moisture profile specimens at the same average moisture content was comparatively smaller, being about a factor of 5 from the value in dry specimens. X-ray radiographic analysis of damage accumulation in compression-compression fatigue revealed interlaminar cracking to be the dominant mode of failure responsible for the observed enhanced cyclic degradation of moisture-conditioned specimens. This finding is corroborated by the observed systematic reduction in interlaminar shear strength as a function of moisture content, which, in turn, increased the propensity for delamination under cyclic compressive loads. Residual strength measurements on cycled specimens indicate significant strength reductions at long lives, particularly in moisture-conditioned specimens.

Tension-compression fatigue studies conducted on a $[0/\pm30]_{3S}$ AS/3501 laminate over a range of negative stress ratios indicated that a strong moisture-induced degradation in fatigue properties can occur even in the presence of moderate compressive components (20% of the total stress range). The observed reductions in N_f in this case were found to be related to the early initiation of fatigue damage in moisture-containing specimens. In tension-tension fatigue of the T300/934, $[0/\pm45/90]_{2S}$ laminate, moisture was found to enhance degradation only slightly. Here, it appears that the influence of moisture is to intensify the susceptibility of this laminate to edge delamination.

INTRODUCTION

The application of advanced graphite/epoxy composites in airframe components offers a high potential for reducing structural weight. Midsized composite structures, such as the DC-10 vertical stabilizer and the L-1011 vertical fin, are in advanced stages of development and flight service evaluation. Design and development of primary wing and fuselage composite structures constitute a necessary follow-up to these efforts. Reliable use of composites in airframe applications calls for, among other things, demonstrated long-term environmental durability of current epoxy-matrix systems such as T300/934 and T300/5208. A vital part of the environmental serviceability program constitutes an assessment of composite performance under static and cyclic loading in high-temperature (to 350° F (170° C)), high-humidity environments. Further, a need exists to evaluate experimentally and analytically the effects of accelerating the environmental spectra on composite performance so as to provide an alternative to real-time environmental testing of composite structures.

The effect of moisture and temperature on the static mechanical properties of epoxy-matrix composites has been the subject of numerous investigations in recent years. It is well established that the resin matrix is plasticized by absorbed moisture, causing a reduction in resin-controlled mechanical properties such as the compressive strength (σ_c), transverse-tensile strength, and in-plane

and interlaminar shear strengths, particularly at elevated temperatures (200°-300° F (93°-149° C)). Table 1 summarizes the findings of several investigators [1-7] in the area of environmental effects on static properties. Review of the table suggests that, under hot-wet conditions, the static properties of the quasi-isotropic layup $[0/\pm45/90]_s$ and other matrix-dominated laminates, such as $[\pm45/0/\pm45_2]_s$, can be significantly affected, whereas those of the strongly anisotropic layups, such as $[0/\pm45/0]_{2s}$, are only moderately affected. The extent of room-temperature strength degradation (both tensile and compressive) of moisture-containing (to 1.5%) laminates is, however, negligibly small for all laminates of practical interest [3,7].

In contrast to the information available about static behavior, data on the effect of environment on the fatigue behavior of graphite/epoxy composites are limited. Generally, fatigue properties are degraded by both moisture and temperature. For example, flexural fatigue results [8] on angle-ply materials demonstrate a significant reduction in fatigue life in the presence of a hot-wet environment, particularly for tension-compression cycling. Most aspects of the environmental-fatigue problem, however, are not well understood. Such aspects include the effect of moisture on the fatigue limit and the failure mode of practical laminates, the role of moisture gradients (as opposed to uniform moisture distribution) on fatigue behavior, the effect of moisture-conditioning cycles on

the subsequent cyclic behavior of the composite, and the evaluation of the rate of fatigue damage accumulation around stress concentrators in the presence of an environment.

This paper describes experimental work undertaken to determine the moisture-induced cyclic response of the T300/934 graphite/epoxy composite system, which is typical of those in current use. The compression-compression-fatigue (C-C-F) behavior of $[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$ laminates as a function of moisture content is first discussed. An important objective of these C-C-F studies was to evaluate the effect of moisture gradient (obtained by an accelerated moisture-conditioning procedure) versus a flat moisture profile (attained through controlled humidity-temperature conditioning to an equilibrium moisture level) on fatigue behavior. The effect of compressive components on the moisture-induced tension-compression-fatigue (T-C-F) behavior of an angle-ply, matrix-dominated, AS/3501 laminate ($[0/\pm30]_{3s}$) is then described. Some results on the tension-tension-fatigue (T-T-F) behavior of the $[0/\pm45/90]_{2s}$ laminate are also presented.

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EXPERIMENTAL

The angle-ply laminates used in this investigation were manufactured by the Lockheed Missiles and Space Company from prepreg tapes of T300/934 (T) and AS/3501 (AS) systems. The systems and the layups of the laminates were as follows:

(1) T300/934—$[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$; and (2) AS/3501—$[0/\pm30]_{3s}$. The fibers used in the tapes were of the high-strength [3200 MN/m^2 (460 ksi)], intermediate modulus [220 GN/m^2 (32×10^6 psi)] variety. Consolidation of the layups was effected in an autoclave at a pressure of 0.7 MN/m^2 (100 psi) and at a temperature of 347° F (175° C) for 2 hours. The resultant sheets, with midplane symmetry, had a fiber content of about 0.63 V_f, a density of 1600 kg/m^3 (0.057 lb/in.^{3}), and a thickness of about 2.5 mm (0.1 in.).

Compression tests and C-C-F tests were performed on specimens of the $[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$ laminates in the dry and moisture-conditioned states, using the Celanese compression fixture (supplied by the Celanese Research Corporation, Summit, NJ). This fixture is similar in design to the one proposed in ASTM Standard D 3410 for compression testing of oriented fiber composites [9]. The as-received specimens contained about 0.5% moisture. Specimens were vacuum-dried at 212° F (100° C) for 10 days to produce "dry" specimens with about 0.1% moisture prior to moisture-conditioning. Tests were performed on specimens in the dry, as-received, 1.4%, and 2% moisture conditions. Parallel-sided coupons provided with fiberglass or aluminum tabs in the grip section were used in the C-C-F studies. The length of the untabbed gage section was 12.7 mm (0.5 in.), the overall specimen length was 127 mm (5 in.), and the specimen width was 6.35 mm (0.25 in.). The C-C-F tests were

performed at a stress ratio (R) of 10 and a frequency (ν) of 10 Hz. In these tests, change in compliance of the specimen was continuously monitored during cycling. In constant displacement tests, the load decay was continuously monitored so that specimens could be examined for microstructural damage at any stage in the load decay process. Cycled, unbroken specimens were subjected to x-ray analysis to identify failure modes and mechanisms. Specimens were treated with tetrabromoethane (TBE) opaque additive prior to x-ray radiography to enhance the image quality.

Moisture-conditioning of the C-C-F specimens was effected in two ways. In the first, specimens were conditioned in an accelerated manner by immersing them in 194° F (90° C) hot water for about 4 days. This treatment resulted in specimens having a 1.4% average moisture content and containing a significant through-the-thickness moisture concentration gradient. The maximum moisture content corresponding to this treatment was experimentally determined to be about 1.8%. In the second, specimens were conditioned in a humidity chamber maintained at 176° F (80° C) and 80% relative humidity for about 2 months. Specimens conditioned by this method also had an average 1.4% moisture content, but had no significant moisture gradients. This was inferred by the negligible increase in specimen weight that occurred over a 2-week period preceding the end of the 2-month conditioning period, signifying equilibrium moisture pick-up conditions. The moisture conditioning treatments are schematically represented in

Figure 1. Specimens with 2% moisture content were conditioned in an accelerated manner by immersion in 194° F (90° C) hot-water for 4 weeks and then immersion in cold water for 1 week. These specimens presumably had no moisture gradients in them since this composition (2%) represents the moisture saturation limit in this material. The 1.4% moisture series specimens were kept under refrigeration at 36° F (2° C) until ready to use (1 to 2 weeks) so as to prevent any moisture loss from specimens and preclude change of concentration profile. The longest test conducted was about one week at room temperature, during which time moisture loss from these specimens was found to be insignificant. The 2% moisture specimens were stored under water at 77° F (25° C) until test time. These specimens lost moisture very rapidly at room temperature and, therefore, an effort was made to minimize moisture loss by providing a wet cotton pack around the specimen gage section through the test period.

Most of the specimens used in the C-C-F test program were moisture-conditioned in the untabbed condition so that weight gains could be precisely determined. They were subsequently tabbed with aluminum or 0°/90° cross-ply fiberglass tabs prior to testing. Hysol 9309 room-temperature cure adhesive, or a commercial super-bonder, was used for bonding the tabs to the specimens. A number of specimens were moisture-conditioned in hot water directly in the as-tabbed condition. However, in this case, specially fabricated 10° off-axis fiberglass tabs had to be used, together with a

protective heavy coating of silicone rubber in the tab-region prior to hot-water immersion. These procedures were necessary to help circumvent recurrent tab failures during cyclic testing of moisture-conditioned $[0/\pm45/0]_{2S}$ specimens. These techniques of tabbing and conditioning nevertheless were only partially successful.

Tension-compression-fatigue tests were conducted on AS/3501 specimens of the $[0/\pm30]_{3S}$ laminate moisture-conditioned at 140° F (60° C) and 80% RH for 4 weeks and that contained about 1% moisture. This treatment presumably resulted in significant through-the-thickness moisture gradients since the maximum moisture content expected under these conditions is about 1.5%. The overall fatigue behavior of this laminate in the dry condition was characterized earlier [10]. Unsupported coupon specimens with tapered fiberglass end tabs, measuring 150 mm (6 in.) by 9.5 mm (0.38 in.) by 2.5 mm (0.1 in.), were used in these studies. The specimens had a gage section of 37 mm (1.5 in.). Compression loads used in the T-C-F tests were in the linear region of the stress-strain curve in compression. Limited tension-tension fatigue (T-T-F) studies were also performed on the $[0/\pm45/90]_{2S}$ laminate in the dry and moisture-conditioned (1.4%) states, mainly for comparison with the corresponding C-C-F results. The moisture-conditioning treatment employed for the T-T-F specimens was identical to the accelerated conditioning treatment utilized for the C-C-F specimens (194° F (90° C) hot-water immersion). The conditioning period was different, however, because of the differences in geometry of the specimen in the two cases.

The form of the through-the-thickness moisture gradient existing in specimens subjected to the accelerated moisture-conditioning treatment (194° F (90° C) hot water for 4 days) was approximated by using the theory proposed by Shen and Springer [11]. Since the edge surface area was a significant fraction (0.3) of the total surface area for these specimens, and the edges were not provided with a coating impermeable to moisture during conditioning, it was essential to consider moisture diffusion through all six faces of the specimen. The overall diffusivity, D, in such a case, corrected for edge effects for a laminated composite, is given in Reference [11]. This expression was used to compute D for the $[0/\pm45/90]_{2s}$ laminate specimens using the value of resin-diffusivity at 194° F (90° C) [11], and found to be 1.38×10^{-6} mm^2/sec. This value of D predicted quite accurately the time required for a given moisture absorption. Using the classical solution to Fick's Second Law and calculated diffusivity, the moisture concentration profile at the end of the conditioning period was determined (as shown in Figure 2). This figure illustrates a high moisture concentration in the outer surface plies and a fairly sharp decrease, to about 55% of the maximum value, at the center of the specimen.

The standard short-beam shear (SBS) test method [12] was used to determine the apparent interlaminar shear strength (ILSS) of the $[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$ laminates in the dry and moisture-conditioned states. A span/depth ratio of 4 produced consistent

horizontal shear failures in both the dry and wet conditions. A minimum of 20 specimens were tested for most of the conditions to evaluate degradation in the ILSS. Short-beam shear tests were also conducted on a $[0]_{16}$ laminate for comparison. For this laminate, however, a span/depth ratio of 6 was necessary to produce shear failures in the moisture-conditioned states.

RESULTS

Compressive Strength

The strength in compression (σ_c) of the $[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$ laminates, measured by the Celanese method, was 15% greater than the corresponding strengths in tension. Scatter in σ_c about the mean value was generally within ±7.5% for both laminates in both the dry and the wet conditions. The variation of σ_c with moisture content for these laminates is shown in Figure 3. The reduction in σ_c up to 1.4% moisture (gradient/no gradient) is small compared with that observed at 2% moisture, where σ_c decreases by about 20% for both laminates. The observed variation in σ_c signifies increasingly strong degradation in matrix properties in the range of 1.4 to 2% moisture.

Compression-Compression-Fatigue

S-N Curves- S-N ($\Delta\sigma$ vs log N_f) curves for the T300/934, $[0/\pm45/90]_{2s}$ laminate as a function of moisture content are

presented in Figure 4. The results in Figure 4 are cross-plotted in Figure 5 (log N_f vs % moisture) at an arbitrary stress level to illustrate in clearer terms the moisture-induced degradation in the C-C-F behavior of this laminate. With increasing moisture, fatigue life for a given stress amplitude decreases. The dry (0.1% moisture) and as-received (0.5% moisture) conditions result in nearly similar lives and are therefore treated as equivalent. The extent of reduction in fatigue life at 1.4% moisture content is significant and is essentially independent of applied stress. A reduction by a factor of 4 to 5 (7 to 8 for the gradient case) in the average fatigue life occurs at this moisture level. This finding is particularly important in view of the fact that, for this moisture content (1.4%), the strength in compression is virtually unaffected for this laminate. Therefore, strong moisture-induced degradation in compressive-fatigue properties can occur in the absence of a similar degradation in compressive strength. Fatigue tests at 2% (saturation) moisture level reveal even further reductions in N_f.

A comparison of S-N curves for the gradient and nongradient cases at 1.4% moisture content (Figure 4) reveals that the gradient specimens, at all stress levels, are subject to nearly double the reduction in fatigue life noted for corresponding nongradient specimens. From this observation, it can be surmised that accelerated moisture conditioning leads to a conservative estimate of fatigue life.

S-N curves for the T300/934, $[0/\pm45/0]_{2s}$ laminate as a function of moisture content are presented in Figure 6. These curves are essentially similar in form to those for the $[0/\pm45/90]_{2s}$ laminate (Figure 4). A reduction by a factor of 7 to 8 in average fatigue life occurs for gradient specimens containing a moisture content of 1.4%. Stronger reductions in N_f are evidenced at a moisture content of 2%. Therefore, it appears that both these laminates exhibit almost identical behavior under C-C-F as a function of moisture content. The endurance limit at 10^7 cycles for the dry and as-received conditions occurs at about 40 and 50% of the respective average ultimate compressive strengths for the $[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$ laminates. This apparent fatigue limit is clearly shifted to lower stress levels with increasing moisture in both laminates; the amount of reduction appears to be about 10% for a moisture content of 1.4% and 15-20% for a moisture content of 2%.

Failure modes and mechanisms- An extensive x-ray radiographic study of fatigue-damaged specimens was undertaken in an effort to determine the failure modes and mechanisms operative in both the dry and moisture-conditioned specimens. Specimens were cycled to various fractions of the average life at a given stress level or, alternatively, until a given change in compliance had occurred, then removed from the test machine and subjected to x-ray

radiography. Additionally, a number of run-out specimens that had survived in excess of 4×10^6 cycles were also examined.

X-ray radiographs displaying interlaminar cracks in representative specimens of $[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$ laminates are presented in Figures 7 and 8, respectively. These photographs represent edge views of specimens whose moisture content, moisture distribution, cyclic history, and stress amplitude are detailed beneath the respective radiographs. It is apparent from these figures that multiple interlaminar cracks occur in all moisture-conditioned specimens and are essentially independent of cyclic stress amplitude and laminate orientation. In contrast, the dry specimens, containing only 0.1% moisture, reveal at best a single delamination crack near failure (Figure 7(a)) or, more often, small segments of such cracks (Figures 7(a), 8(b)). A natural tendency for delamination is expected under C-C cycling, due to Poisson effects. However, very few delamination cracks are found in dry specimens, and are especially nonexistent at low cyclic stress levels. These observations suggest that in dry or as-received specimens, failure mechanisms other than delamination play a primary role in effecting failure. In summary, a change in failure mode occurs in dry and wet specimens, namely, from general failure mechanisms in dry specimens to predominantly interlaminar cracking in moisture-conditioned specimens.

A careful study of interlaminar cracks in a large number of moisture-conditioned fatigue-damaged specimens revealed that, for the $[0/\pm45/90]_{2s}$ laminate, about 60% of the cracks occur between +45°/-45° plies and about 20% between 90°/±45° plies and between 0°/±45° plies. An occasional crack between the central 90°/90° plies was also observed. In case of the $[0/\pm45/0]_{2s}$ laminate, on the average, 50% of the cracks occurred between 0°/±45° plies and 50% between +45°/-45° plies. These cracks initially formed in the outer plies, particularly in specimens that contained a moisture gradient (Figure 7(e)), due presumably to the higher moisture concentration in outer plies, but subsequently appeared in the inner plies as well. In addition, multiple splits in surface 0° plies were frequently observed and then followed by delamination of plies close to the surface.

Other failure mechanisms found to occur in moisture-conditioned specimens are shown in the x-ray radiographs of Figure 9 (front faces of specimens). Figure 9(a) illustrates the nature of damage in a $[0/\pm45/90]_{2s}$, 2% moisture-conditioned specimen approaching failure at 40% σ_c. Intense damage along ±45°, 0°, and 90° directions (dark lines in the photograph) and patches of delamination can be discerned. The former type of damage signifies fiber-matrix separation, due to impairment of the interfacial bond between the matrix and the fibers. Figure 9(b) depicts similar damage in a $[0/\pm45/0]_{2s}$, 1.4% moisture specimen after 4×10^6 cycles at 50% σ_c.

The nature of fatigue damage occurring in dry specimens was significantly different, as has already been remarked. Figure 10 illustrates damage seen from the front faces of representative dry specimens of the two laminates. Figure 10(a) demonstrates stepwise cracking in the outer 0° ply and failure in one set of 45° directions in a $[0/\pm45/90]_{2s}$ dry (0.1% moisture) specimen after 1.5×10^5 cycles at 50% σ_c. Usually, at this point just preceding failure, a single delamination crack in the outer surface plies between 0°/45° ply or +45°/-45° ply appeared. This mechanism of failure was also observed in some $[0/\pm45/0]_{2s}$ dry specimens. Figure 10(b) reveals fine, general failure in ±45° directions occurring in the entire gage section of a $[0/\pm45/0]_{2s}$ dry (0.1% moisture) specimen after 5×10^6 cycles at 55% σ_c. This type of damage is similar to that observed at long lives in T-T cycling of the same laminate [13].

Interlaminar shear strength- The abundance of interlaminar cracks observed in fatigue-tested, moisture-containing specimens suggests the possibility of interlaminar weakening due to moisture absorption. Therefore, the apparent interlaminar shear strengths of both laminates were determined as a function of moisture content. Results of the short beam shear tests are presented for the $[0/\pm45/90]_{2s}$ laminate in Figure 11, where it is seen that the ILSS progressively decreases with increasing moisture content. Similar decreases with increasing moisture were observed for the $[0/\pm45/0]_{2s}$

and $[0]_{16}$ laminates as well, but are not shown. On the average, 25 and 15% reductions in ILSS occurred at 1.4% moisture content for the $[0/\pm45/90]_{2s}$ (both with and without a moisture gradient) and the $[0/\pm45/0]_{2s}$ laminates. These reductions in ILSS in the presence of 1.4% moisture appear to be directly responsible for promoting interlaminar cracking under cyclic compressive loads.

Residual strengths- The residual compressive strengths (σ_r) of a variety of cycled specimens were determined in an attempt to assess the degradation in composite compressive strength after a given history of cycling. The observed values are given in Tables 2 and 3 for the $[0/\pm45/90]_{2s}$ and the $[0/\pm45/0]_{2s}$ laminates. Also shown are the corresponding cyclic stress level, moisture content, and number of loading cycles given each specimen. Examination of the data in these tables discloses that, in general, considerable strength reductions occur in moisture-containing specimens subjected to C-C cycling. Considering the high-endurance specimens (4×10^6 to 10^7 cycles range), it is seen that, for the $[0/\pm45/90]_{2s}$ laminate, a 35% reduction in the original σ_c occurs after 5×10^6 cycles at $\sigma_{max} = 35\%\ \sigma_c$ in the wet (1.4% moisture) condition. The as-received (0.5% moisture) specimen exhibits a 20% reduction in σ_c after 6.7×10^6 cycles at $\sigma_{max} = 40\%\ \sigma_c$, whereas the corresponding dry specimen exhibits virtually no strength degradation after 5×10^6 cycles. A similar strong (40%)

reduction in σ_c occurs in a moisture-conditioned (1.4% moisture) specimen of the $[0/\pm45/0]_{2s}$ laminate after 4×10^6 cycles at $\sigma_{max} = 50\%\ \sigma_c$. For this laminate, the corresponding as-received specimens exhibit 15-25% reductions in σ_c in the range of 5×10^6 to 10^7 cycles.

These observations suggest that the laminates investigated are susceptible to significant strength degradation at long lives both in the dry and as-received conditions. This effect appears to be accentuated by moisture, with resultant considerably lowered residual strengths. The larger strength reductions evidenced in moisture-containing specimens may be associated with the occurrence of multiple interlaminar cracks in these specimens.

Tension-Compression-Fatigue

S-N curves- The effect of moisture on the T-C-F behavior of the AS/3501, $[0/\pm30]_{3s}$ laminate was investigated. The fatigue behavior of this laminate in the dry condition has been characterized in an earlier study [10]. Varying amounts of compressive components were added to a constant tensile component (60% of the tensile strength) to produce negative stress ratios in the range of about -0.1 to -0.7. The compressive components were always within the limit of linearity of the stress-strain curve in compression for the unrestrained test specimens. T-C-F tests were performed at five different negative R values in the range cited. The

results are plotted in terms of stress range ($\Delta\sigma$) versus log N_f in Figure 12 for the dry specimens (0.1% moisture) and for specimens containing 1% moisture. A definite reduction in fatigue life occurs for the moisture-conditioned specimens at all values of R investigated. Of importance is the observation that a significant moisture-induced degradation in N_f occurs even in the presence of a relatively small compressive stress component (20% of σ_c).

<u>Compliance decay</u>- For a number of specimens in this study, load-displacement hysteresis loop changes were recorded as a function of cycles. For the moisture specimens, a 15-20% change in slope of the initial hysteresis loop occurred relatively early in life (<20% N_f). Subsequent changes in slope were gradual until close to specimen failure. For the dry specimens, comparable slope changes occurred much later in life ($\sim$ 75% N_f), as detailed in Reference [10]. It therefore appears that loss in specimen stiffness and initiation of fatigue damage commence much earlier in moisture-conditioned specimens with resultant reduced life.

Tension-Tension-Fatigue

Limited tension-tension-fatigue (T-T-F) studies were performed on the T300/934, $[0/\pm45/90]_{2s}$ laminate in the dry (0.1% moisture) and 1.4% moisture conditions to provide a comparison with the C-C-F results. S-N curves at $R = 0.1$ are presented in Figure 13. These data indicate a reduction, by a factor of 2 to 3, in average fatigue

life due to moisture at either stress level considered. The fatigue life of the $[0/\pm45/90]_{2s}$ laminate under T-T cycling is influenced by edge effects, with failure of this laminate occurring inevitably through extensive edge delamination [13]. It appears that moisture accelerates the onset of edge delamination (due to strong reduction in ILSS at 1.4% moisture content as shown in Figure 11) and thereby causes a lowering of fatigue life. The results, however, indicate that the extent of reduction in fatigue life due to moisture is far less under T-T cycling than under C-C cycling, despite the edge delamination problem prevalent in the former mode of cycling.

DISCUSSION

The results of static compression tests of the present study (Figure 3) are in general agreement with the findings of other investigators [3,5]. It can be stated that moisture contents up to 1.5% have a negligible effect on the room-temperature compressive strengths of T300/934, $[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$ laminates. The increased degradation in σ_c in the 1.5-2% moisture range may at least in part be related to reversible matrix degradation resulting from prolonged high-temperature exposure (200° F (93° C)) during moisture conditioning. Plasticization of the matrix apparently occurs in this moisture range since, on drying fully saturated specimens, the original dry σ_c value is restored. The matrix

degradation is reflected as a strong decrease in the apparent ILSS in the range of 1-2% moisture (Figure 11) and, since the ply compressive strength depends on the ILSS [14], σ_c of the laminates exhibits a significant decrease in this moisture range as well. The reduction in the apparent ILSS with moisture content, however, appears to be more severe than the corresponding reduction in σ_c. The relationship between ILSS and σ_c does not appear to be a linear one under moisture-degradation conditions.

The C-C-F results indicate that moisture contents in the vicinity of 1.5% can cause substantial reductions in fatigue life at any stress level (Figures 4 and 6) for the widely different laminates (quasi-isotropic $[0/\pm45/90]_{2s}$ and fiber-dominated $[0/\pm45/0]_{2s}$) investigated. Surprisingly, both laminates exhibit similar C-C-F behavior as a function of moisture. This similarity was attributed to interlaminar weakening due to moisture absorption, a feature that should be common to all multidirectional laminates. It therefore seems reasonable to assert that any multidirectional graphite/epoxy is likely to exhibit a moisture-induced degradation in C-C-F behavior similar to that observed for the laminates in this study. About an order-of-magnitude reduction in N_f is evidenced at 1.4% moisture for both laminates, with a resultant ∿10% reduction in the apparent fatigue limit. This finding is all the more significant in view of the fact that strong reductions in N_f occur, despite a negligible reduction in σ_c for either laminate at this moisture

content. Further, a 20% reduction in σ_c at 2% moisture does not appear to cause exceedingly large reductions in N_f over that observed at 1.4% moisture for the $[0/\pm45/90]_{2s}$ laminate (Figure 5). These observations suggest that any change in σ_c due to moisture is perhaps not an indicator of potential fatigue resistance. Another important finding arising from the C-C-F studies is that accelerated moisture-conditioning results in a lower estimate of fatigue life. This was shown by comparing the fatigue behavior of specimens with and without moisture gradients at equivalent moisture content (Figure 4). A reasonable explanation for the observed effect is that the higher moisture concentration in the outer surface plies in gradient specimens (Figure 2) encourages interlaminar cracking between these plies relatively early in life, with subsequent reduction in fatigue life.

The observed strong reductions in N_f at high moisture contents (1.4% and greater) is directly related to the correspondingly large reductions in ILSS at these moisture contents. This conjecture is borne out by the fact that interlaminar cracking is the primary mode of failure observed in moisture-containing specimens subjected to C-C-F. Therefore, it appears that increases in ILSS of graphite/epoxy laminates through manufacturing or other means would effectively improve their resistance to C-C-F in the presence of 1% or greater moisture content.

The apparent fatigue limit (10^7 cycles) in C-C-F is found to be about 45-50% σ_c for the $[0/\pm45/0]_{2s}$ laminate in the as-received condition (0.5% moisture). This is considerably lower than the corresponding fatigue limit in T-T-F, which is about 65-70% of the tensile strength [13]. Moreover, the presence of about 1.5% moisture is seen to further decrease the C-C fatigue limit by about 10% to a value of 35-40% σ_c. Whether these results can be applied to larger panels representative of structural components remains to be determined. Nevertheless, the reported occurrence of both static and fatigue failures in the compression skin of real structural components [6] emphasizes the importance of compressive loads in composite performance. Similar considerations apply to quasi-isotropic laminates as well.

Extensive edge delamination was observed in the $[0/\pm45/90]_{2s}$ laminate under cyclic tensile loads, making it difficult to define a T-T fatigue limit without ambiguity. The effect of moisture on the T-T-F behavior of this laminate is seen to be slight and presumably is related to moisture-enhanced edge delamination. The effect of moisture under T-C-F is seen to be as significant as under C-C-F, even in the presence of only moderate compressive stress components. Similar moisture-induced degradation of angle-ply graphite/epoxy has been observed in flexural fatigue tests at $R = -1$ [8]. The major mode of failure in moisture-conditioned specimens subjected to C-C-F was shown to be

delamination between plies. This mode of failure apparently overshadows the effects of other possible local failure modes.

CONCLUSIONS

The results of the present investigation disclose that moisture causes a significant degradation in the compression-compression-fatigue behavior of the T300/934, $[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$ graphite/epoxy composite laminates. The form of moisture distribution existing in specimens (gradient or flat profile) is shown to be an important factor in determining fatigue life under compression-compression cycling. A factor of 7 to 8 reduction in N_f occurs for gradient specimens containing an average moisture content of 1.4%; the corresponding reduction in N_f for the flat moisture profile specimens was only a factor of 4 to 5 from the value in dry specimens. It is apparent from these results that accelerated moisture-conditioning leads to a conservative estimate of fatigue life. Further, it is significant that these large reductions in N_f occur despite a negligible reduction in compressive strength at a moisture content of 1.4%. X-ray radiographic analysis has revealed interlaminar cracking to be the primary mode of failure occurring in moisture-conditioned, fatigue-tested specimens of both laminates; this may be related to the observed strong reduction in interlaminar shear strength at high moisture contents. Significant moisture-induced degradation in fatigue life has also

been observed under tension-compression-fatigue of the AS/3501, $[0/\pm 30]_{3s}$ laminate. Moisture, however, does not appear to have a significant influence on the tension-tension-fatigue behavior of the T300/934, $[0/\pm 45/90]_{2s}$ laminate.

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Table 1. Effect of Environment on Static Properties of Graphite/Epoxy.

Ref.	System	Laminates	Tests	% Moisture/ temp.	Conclusions
1	T300/934	$[0/\pm45/90]_s$ facesheets	Sandwich beam	0 to 1%	σ_c and $\tau_{in\text{-}plane}$ not affected at 75° F } influence of 1% moisture
			Rail shear	75° to 350° F	σ_c and τ reduced substantially at 350° F } influence of 1% moisture
2	T300/5208	0°	Flexure	1.5%	ILSS is lowered by 30% } compared with RTD[a] condition
			SBS	170° F	Modulus of the resin is lowered } compared with RTD[a] condition
3	T300/5208	$[0/\pm45/90]_s$	Compression	Water boil for 24 hr	Moisture strongly affects σ_c at high temperatures (>200° F)

[a]RTD = Room temperature dry.

Table 1. Continued.

Ref.	System	Laminates	Tests	% Moisture/ temp.	Conclusions
4	T300/5208	$[0/\pm45/90]_s$	Pin-bearing	1.5%	Hot-wet pin-bearing strength
		$[0/\pm45]_s$		260° F	is reduced 60% compared with
					RTD value
5	T300/5208	$[0/\pm45/0]_s$	Compression	1.5%	% decrease in σ_c from RTD
	T300/934	L_1 = 15% 0°, 85% 45°		180° F	value: 5208 934
		L_2 = 50% 0°, 50% 45°			L_1 20 24
					L_2 1.5 9
6	T300/5208	$[0/\pm45/0]_s$	Static	2%	250° F wet = 51% RTD predic-
			(bending	250° F	tion. 250° F dry = 47% RTD
			and torsion		prediction. Failure at 250° F
			of box		not dependent on moisture
			beams)		content.

Table 1. Concluded.

Ref.	System	Laminates	Tests	% Moisture/ temp.	Conclusions
7	T300/1034	0° $[0/\pm45/90]_s$ 90°	Tension	0 to 1.5% -165° to 300° F	0° and $[0/\pm45/90]_s$ laminates slightly affected at moisture contents > 1%. 90° laminate was strongly affected.

Table 2. Residual Compressive Strengths: $[0/\pm45/90]_{2s}$ Laminate.

Stress level (% σ_c)	Moisture content (% wt)	N (cycles)	σ_r (MN/m^2)	Decrease from dry σ_c (%)
50	0.1	2.5×10^6	496	24
	0.5	1.5×10^5	590	10
	0.5	1.0×10^5	669	0
	0.5	1.0×10^5	600	8
	1.4[a]	3.5×10^4	538	18
	↓	1.0×10^3	607	7
	↓	5.0×10^3	655	0
	↓	1.3×10^4	538	18
	↓	3.4×10^4	386	41
	↓	3.8×10^4	476	27
	↓	2.5×10^4	503	23
	↓	4.5×10^4	386	41
45	0.1	1.0×10^6	627	4
	0.5	1.2×10^5	627	4
40	0.1	4.6×10^6	627	4
	0.5	6.7×10^6	524	20
	2.0	5.1×10^5	600	8
	↓	6.0×10^5	648	1
	↓	8.4×10^5	517	21
	↓	3.2×10^5	427	35

Table 2. Concluded.

Stress level (% σ_c)	Moisture content (% wt)	N (cycles)	(MN/m^2)	Decrease from dry σ_c (%)
40	2.0	1.6×10^5	579	12
(cont)	2.0	9.0×10^5	360	45
35	1.4[a]	5.0×10^6	427	35
	1.4	5.1×10^6	421	36
	2.0	2.4×10^6	476	27

[a]Moisture gradient.

Table 3. Residual Compressive Strengths: $[0/\pm 45/0]_{2s}$ Laminate.

Stress level (% σ_c)	Moisture content (% wt)	N (cycles)	σ_r (MN/m^2)	Decrease from dry σ_c (%)
55	0.5	5.1×10^6	608	38
	0.5	2.1×10^6	794	19
	1.4[a]	1.5×10^5	724	26
	1.4[a]	4.1×10^5	758	23
50	0.5	1.0×10^7	754	23
	0.5	5.4×10^6	834	15
	1.4[a]	1.2×10^6	738	25
	1.4[a]	4.0×10^6	586	40

[a]Moisture gradient.

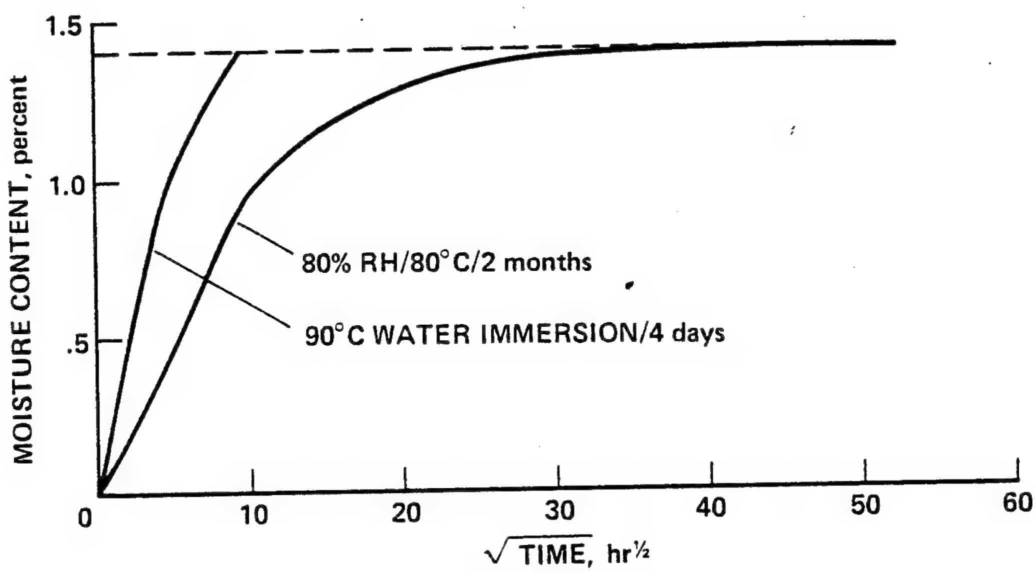

Figure 1. Moisture-conditioning treatments (schematic).

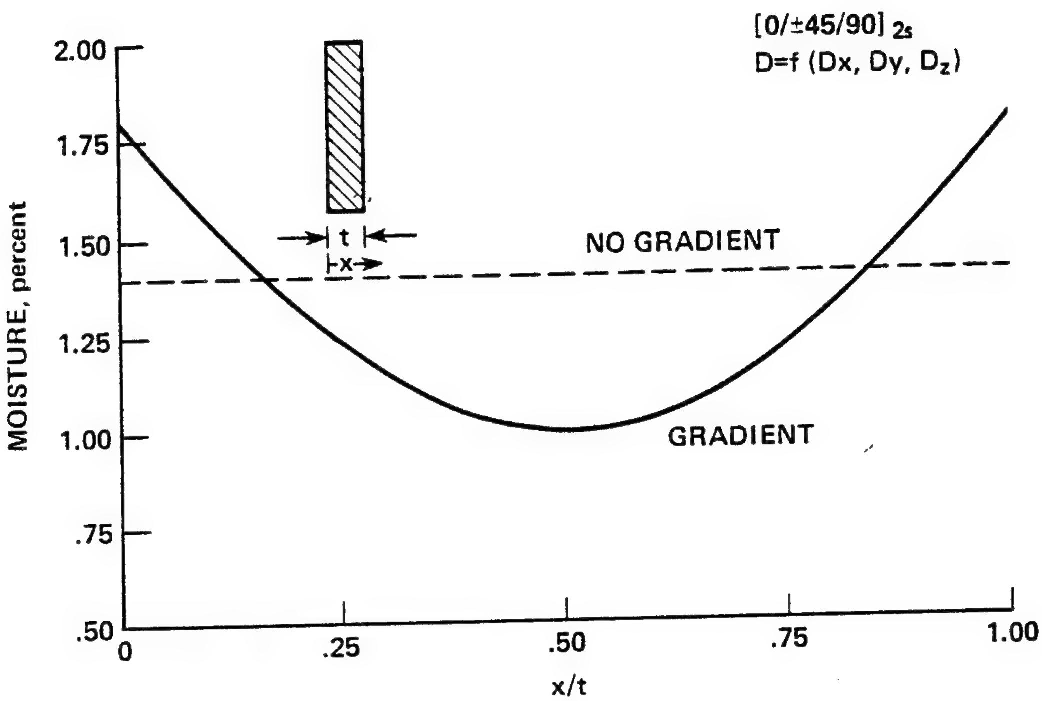

Figure 2. Through-the-thickness moisture concentration gradient in a $[0/\pm 45/90]_{2s}$ compression specimen after 4-day immersion in 90° C water. Profile calculated using the theory proposed in [11].

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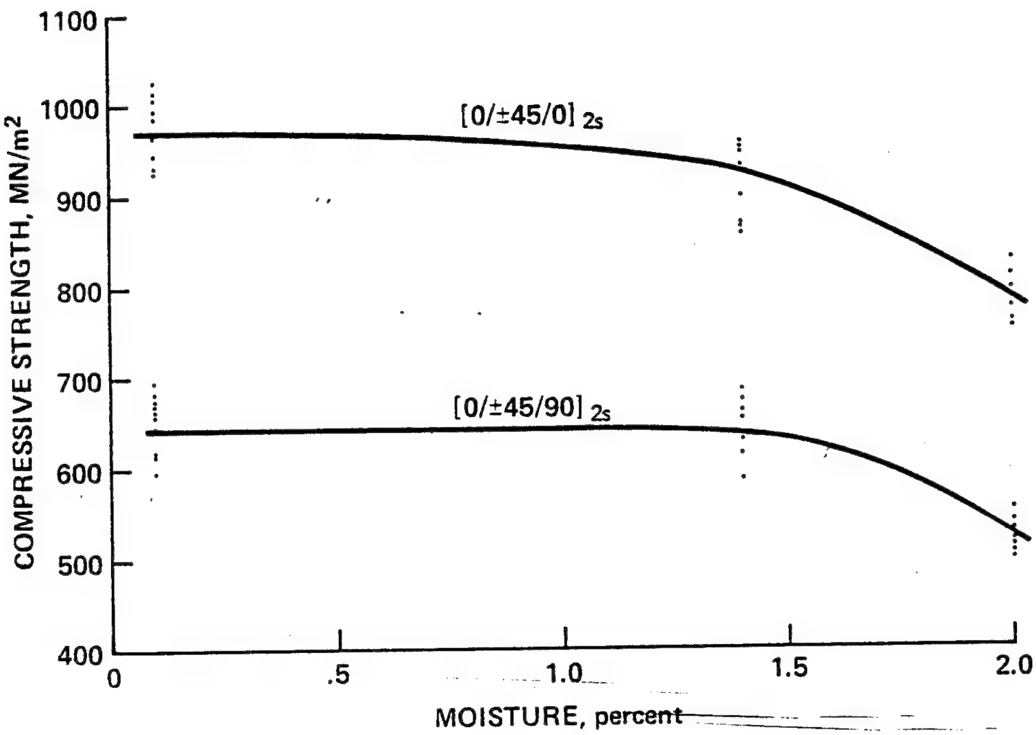

Figure 3. Variation of compressive strength with moisture content for the T300/934, $[0/\pm45/90]_{2s}$ and $[0/\pm45/0]_{2s}$ laminates.

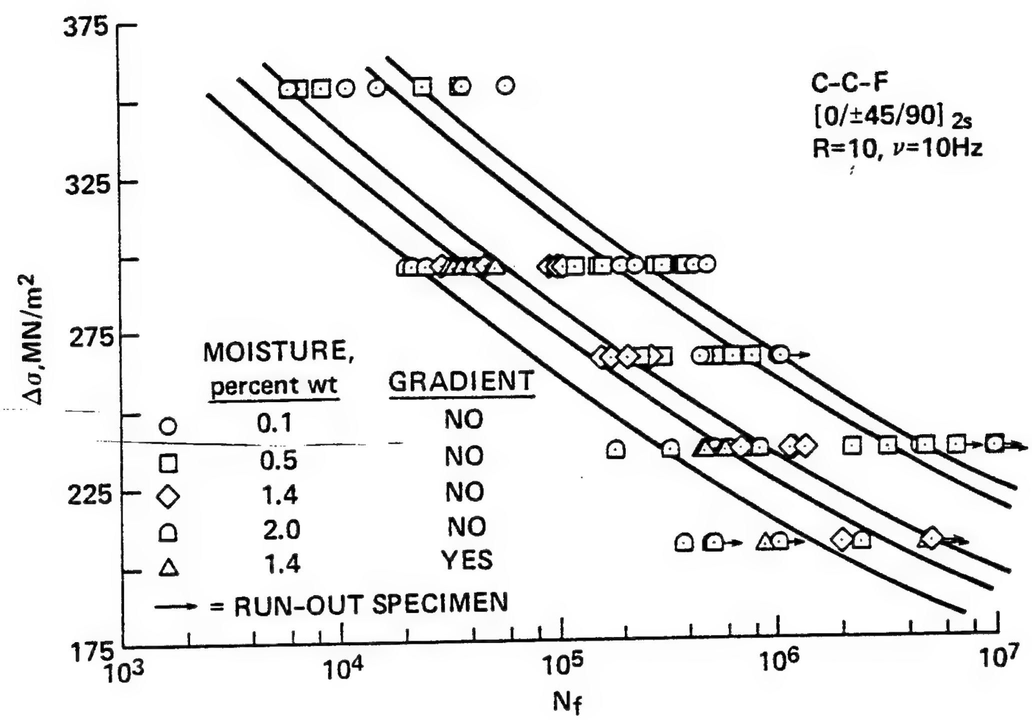

Figure 4. $\Delta\sigma$-log N_f curves for the $[0/\pm45/90]_{2s}$ laminate in the dry and moisture-conditioned states under C-C cycling.

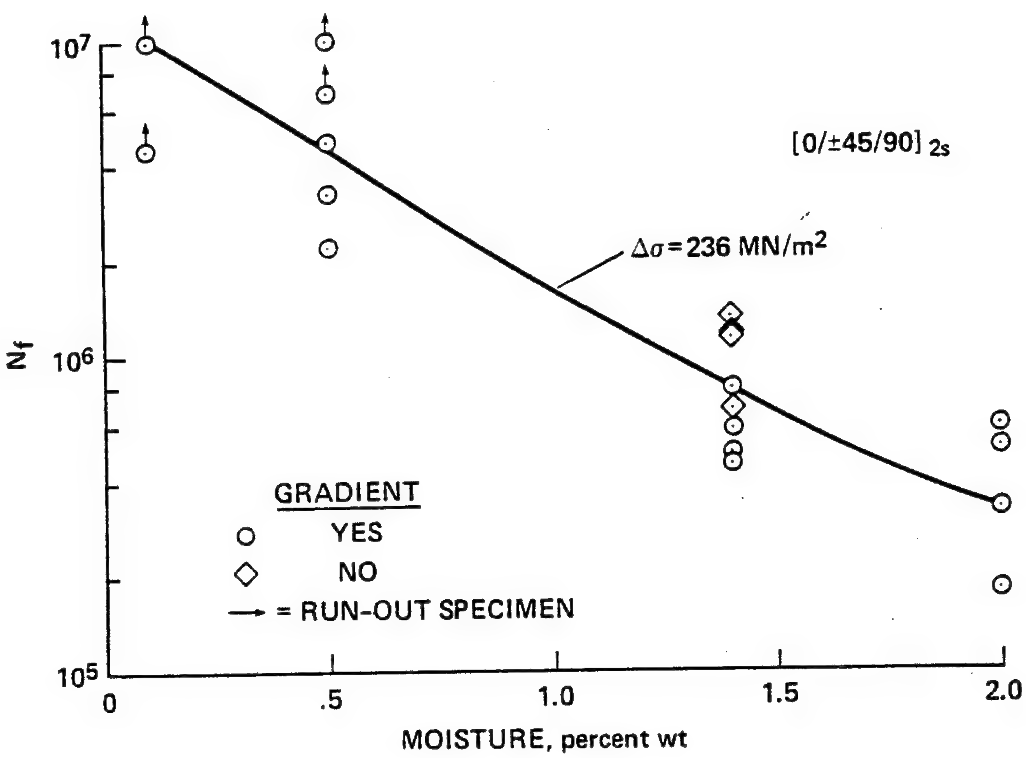

Figure 5. Fatigue life versus moisture content at $\Delta\sigma$ = 236 MN/m^2 for the $[0/\pm45/90]_{2s}$ laminate.

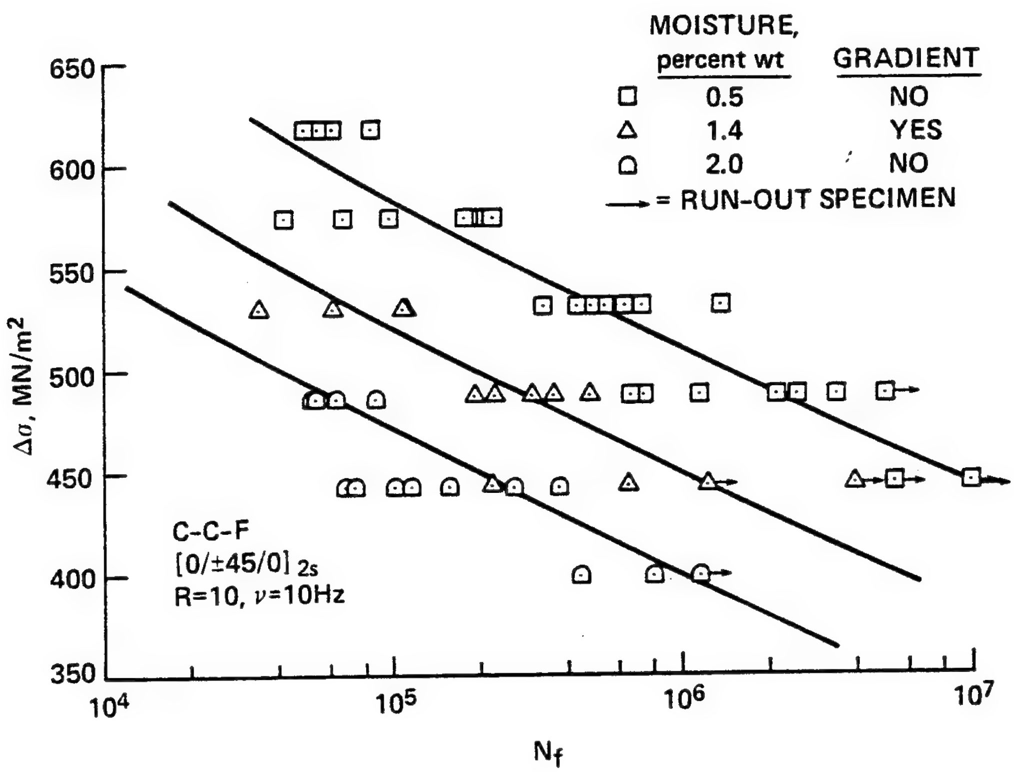

Figure 6. $\Delta\sigma$-log N_f curves for the $[0/\pm45/0]_{2s}$ laminate in the dry and moisture-conditioned states under C-C cycling.

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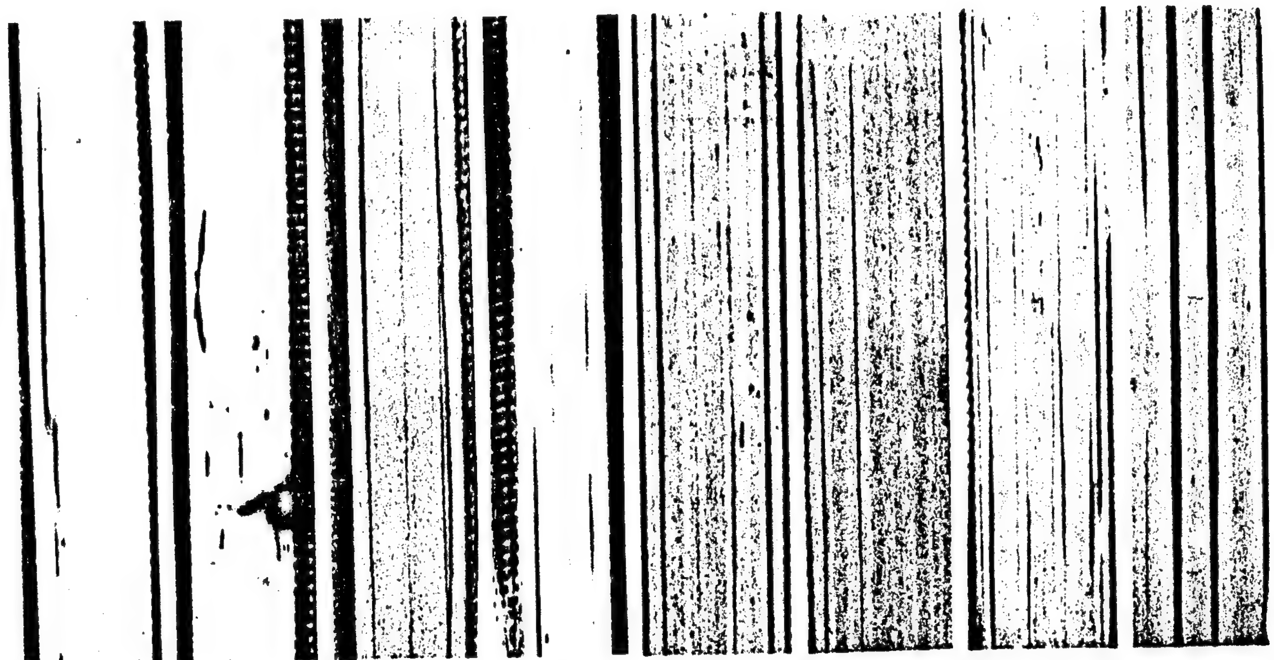
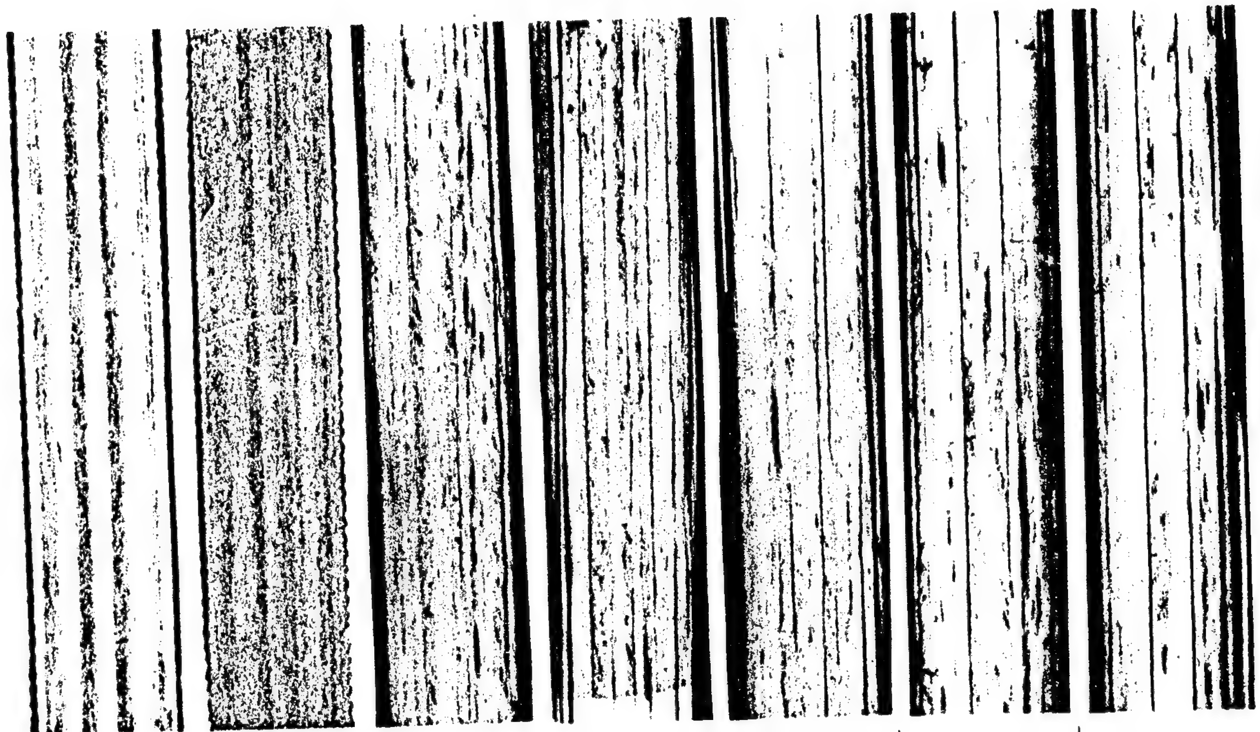
$[0/\pm45/90]_{2s}$ EDGE VIEWS



	a	b	c	d	e	f	g	h
MOISTURE, percent wt	0.1	0.1	1.4	1.4	1.4	2.0	2.0	2.0
GRADIENT	NO	NO	YES	NO	YES	NO	NO	NO
N, cycles	2.5×10^6	4.6×10^6	3.5×10^4	5×10^6	5×10^6	3.2×10^5	8.4×10^5	2.4×10^6
STRESS AMPLITUDE, percent σ_c	50	40	50	35	35	40	40	35

Figure 7. Interlaminar cracks in $[0/\pm45/90]_{2s}$ specimens subjected to C-C-F.

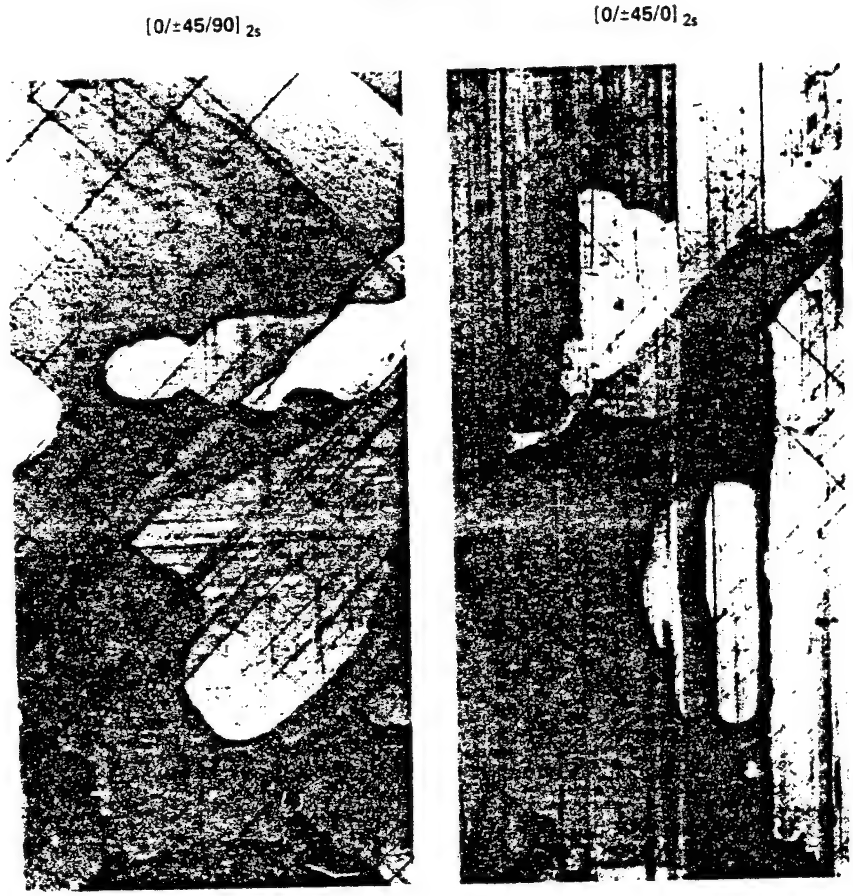
$[0/\pm45/0]_{2s}$ EDGE VIEWS



	a	b	c	d	e	f	g
MOISTURE, percent wt	0.1	0.1	1.4	1.4	1.4	1.4	1.4
GRADIENT	NO	NO	YES	YES	YES	YES	YES
N, cycles	5.1×10^6	10^7	4×10^4	1.5×10^5	4.1×10^5	1.2×10^6	4×10^6
STRESS AMPLITUDE, percent σ_c	55	50	65	55	55	50	50

Figure 8. Interlaminar cracks in $[0/\pm45/0]_{2s}$ specimens subjected to C-C-F.

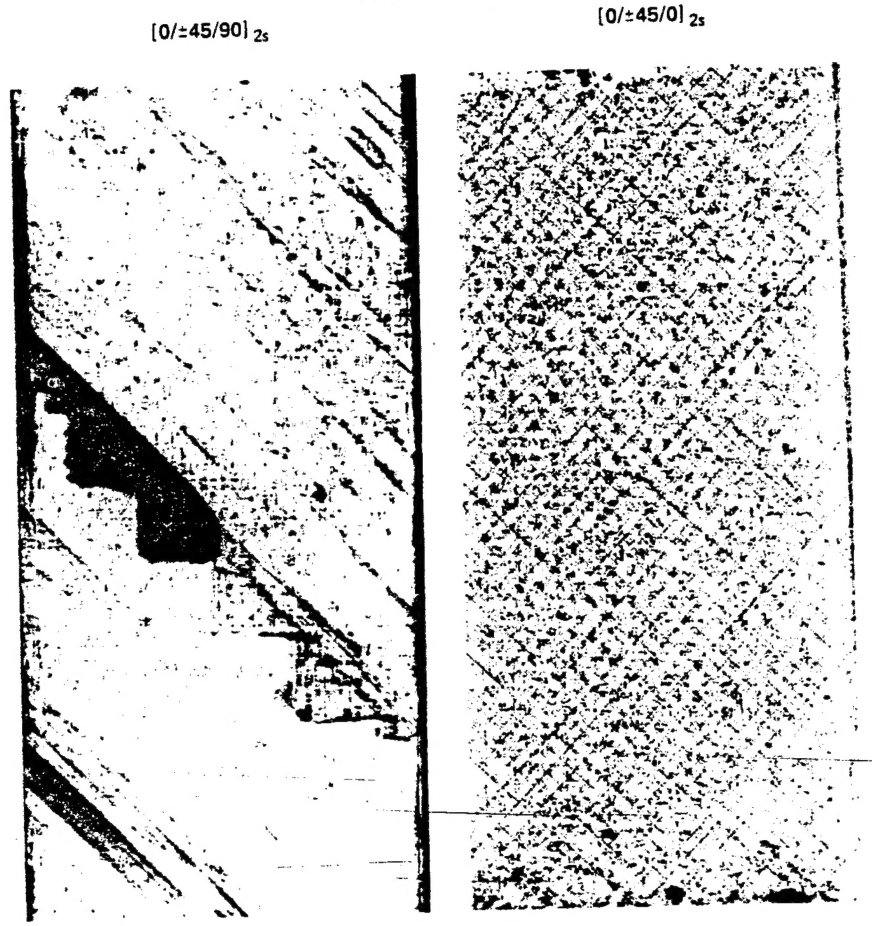
FRONT VIEWS



	a	b
MOISTURE, percent wt	2.0	1.4
GRADIENT	NO	YES
N, cycles	9×10^5	4×10^6
STRESS AMPLITUDE, percent σ_c	40	50

Figure 9. Delamination and cracking along fiber directions in moisture-conditioned specimens subjected to C-C-F.

FRONT VIEWS



	a	b
MOISTURE, percent wt	0.1	0.1
GRADIENT	NO	NO
N, cycles	1.5×10^5	5.1×10^6
STRESS AMPLITUDE, percent σ_c	50	55

Figure 10. Nature of damage observed in dry specimens under C-C cycling.

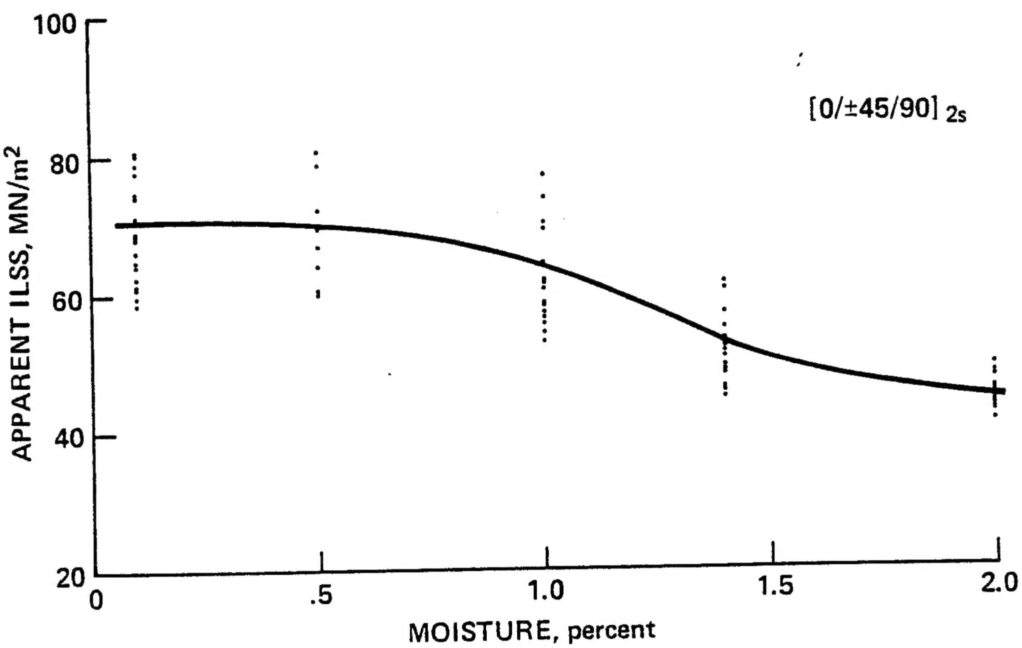


Figure 11. Variation of the apparent interlaminar shear strength with moisture content for the $[0/\pm45/90]_{2s}$ laminate.

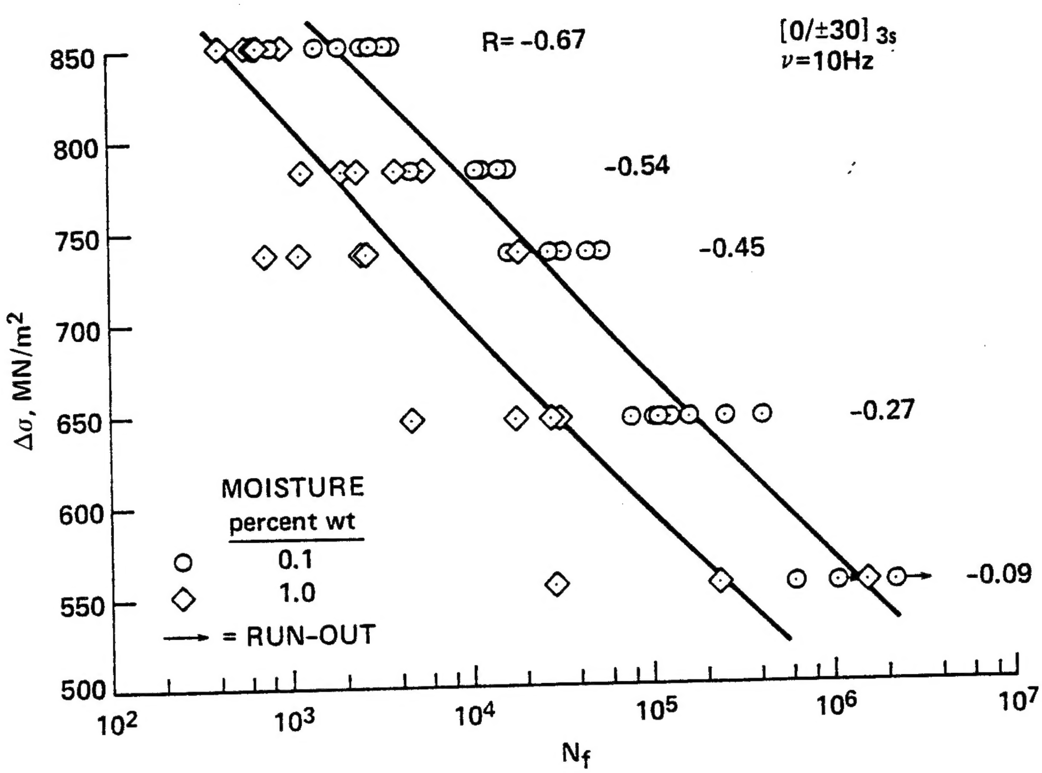


Figure 12. $\Delta\sigma$-log N_f curves for the AS/3501, $[0/\pm30]_{3s}$ laminate in the dry and moisture-conditioned states under T-C cycling.

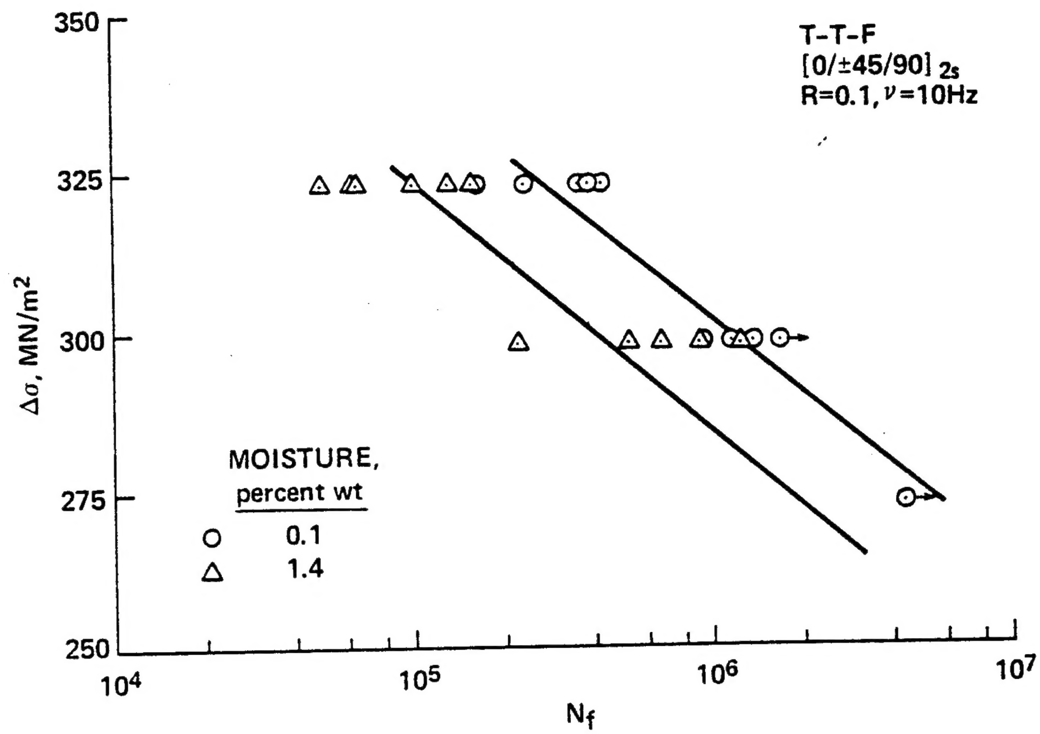


Figure 13. $\Delta\sigma$-log N_f curves for the $[0/\pm45/90]_{2s}$ laminate in the dry and moisture-conditioned states under T-T cycling.